Analytical Laboratory

13339 Hagers Ferry Road
Huntersville, NC 28078-7929
McGuire Nuclear Complex - MG03A2
Phone: 980-875-5245 Fax: 980-875-4349

Order Summary Report

Order Number: **J10090181**

Customer Name(s): Don Scruggs, Robbin Jolly, Ray Lidke, Bill Kennedy

Customer Address: 253 Plant Allen Rd.
Mail Code: ASS01
Belmont,NC 28012

Lab Contact: Jason C Perkins Phone: 704-875-5348

Report Authorized By: (Signature) Digitally signed by JCP7132 (111773) DN: dc=com, dc=duke-energy, dc=ent, dc=nam, ou=Accounts, ou=Personal, ou=PNTransitional, cn=JCP7132 (111773) Date: 2010.10.21 16:28:05 -04'00' **Date:** 10/21/2010

Program Comments:

Allen Test Burn #1: Se Speciation

Sampled: 10/8

Data Flags & Calculations:

Any analytical tests or individual analytes within a test flagged with an "X" or "1" indicate a deviation from the method quality system or quality control requirement. All results are reported on a dry weight basis unless otherwise noted.

Data Package:

This data package includes analytical results that are applicable only to the samples described in this narrative. An estimation of the uncertainty of measurement for the results in the report is available upon request. This report shall not be reproduced, except in full, without the written consent of the Analytical Laboratory. Please contact the Analytical laboratory with any questions. The order of individual sections within this report is as follows:

Job Summary Report, Sample Identification, Technical Validation of Data Package, Analytical Laboratory Certificate of Analysis, Analytical Laboratory QC Reports, Sub-contracted Laboratory Results, Customer Specific Data Sheets, Reports & Documentation, Customer Database Entries, Test Case Narratives, Chain of Custody (COC)

Certification:

The Analytical Laboratory holds the following State Certifications : North Carolina (DENR) Certificate #248, South Carolina (DHEC) Laboratory ID # 99005. Contact the Analytical Laboratory for definitive information about the certification status of specific methods.

Sample ID's & Descriptions:

Sample ID	Plant/Station	Collection Date and Time	Collected By	Sample Description
2010008548	ALLEN	08-Oct-10 10:18 AM	Jeff Womack	FGD Purge Eff
2010008549	ALLEN	08-Oct-10 9:51 AM	Jeremy D.	BioReactor 1 Inf
2010008550	ALLEN	08-Oct-10 9:42 AM	Jeremy D.	BioReactor 2 Eff
2010008551	ALLEN	30-Sep-10 1:45 PM	L. Davis	Metals Trip Blank

4 Total Samples

Technical Validation Review

Checklist:

COC and .pdf report are in agreement with sample totals and analyses (compliance programs and procedures).	☑ Yes	☐ No
All Results are less than the laboratory reporting limits.	☐ Yes	☑ No
All laboratory QA/QC requirements are acceptable.	☑ Yes	☐ No
The Vendor Laboratories have been qualified by the Analytical Laboratory	Yes	

Report Sections Included:

- ☑ Job Summary Report
- ☑ Sample Identification
- ☑ Technical Validation of Data Package
- ☑ Analytical Laboratory Certificate of Analysis
- ☐ Analytical Laboratory QC Report
- ☑ Sub-contracted Laboratory Results
- ☐ Customer Specific Data Sheets, Reports, & Documentation
- ☐ Customer Database Entries
- ☐ Test Case Narratives
- ☑ Chain of Custody
- ☐ Electronic Data Deliverable (EDD) Sent Separately

Reviewed By: Jenny A Herman Date: 10/21/2010

Certificate of Laboratory Analysis

This report shall not be reproduced, except in full.

Order # J10090181

Site: FGD Purge Eff — **Sample #: 2010008548**
Collection Date: 08-Oct-10 10:18 AM — Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	Method	Analysis Date/Time	Analyst
SELENIUM SPECIATION							
Vendor Parameter	Complete				V_AS&C		

Site: BioReactor 1 Inf — **Sample #: 2010008549**
Collection Date: 08-Oct-10 9:51 AM — Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	Method	Analysis Date/Time	Analyst
SELENIUM SPECIATION							
Vendor Parameter	Complete				V_AS&C		

Site: BioReactor 2 Eff — **Sample #: 2010008550**
Collection Date: 08-Oct-10 9:42 AM — Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	Method	Analysis Date/Time	Analyst
SELENIUM SPECIATION							
Vendor Parameter	Complete				V_AS&C		

Site: Metals Trip Blank — **Sample #: 2010008551**
Collection Date: 30-Sep-10 1:45 PM — Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	Method	Analysis Date/Time	Analyst
SELENIUM SPECIATION							
Vendor Parameter	Complete				V_AS&C		

APPLIED SPECIATION
AND CONSULTING, LLC

18804 Northcreek Parkway Bothell, WA, 98011
Tel: (425) 483-3300 Fax: (425) 483-9818
www.appliedspeciation.com

October 17, 2010

Jay Perkins
Duke Energy Analytical Laboratory
Mail Code MGO3A2 (Building 7405)
13339 Hagers Ferry Rd.
Huntersville, NC 28078
(704) 875-5245

Project: Allen – FGD Alternate Fuels Test Burn #1 - Se (LIMS # J10090181)

Dear Mr. Perkins,

Attached is the report associated with three (3) aqueous samples and one associated blank submitted for selenium speciation analysis on October 12, 2010. The samples were received on October 13, 2010 in a sealed cooler at 0.2^{o}C. Selenium speciation analysis was performed via ion chromatography inductively coupled plasma dynamic reaction cell mass spectrometry (IC-ICP-DRC-MS). Any analytical issues associated with the analysis are addressed in the following report.

If you have any questions, please feel free to contact me at your convenience.

Sincerely,

Russell Gerads
Vice President
Applied Speciation and Consulting, LLC

Applied Speciation and Consulting, LLC

Report prepared for:

Jay Perkins
Duke Energy Analytical Laboratory
Mail Code MGO3A2 (Building 7405)
13339 Hagers Ferry Rd.
Huntersville, NC 28078

Project: Allen – FGD Alternate Fuels Test Burn #1 - Se (LIMS # J10090181)

October 17, 2010

1. Sample Reception

Three (3) aqueous samples and one associated blank in 125mL HDPE bottles (provided by Applied Speciation and Consulting) were submitted for selenium speciation analysis on October 12, 2010. The samples were received on October 13, 2010 in a sealed container at 0.2^{o}C.

The samples were received in a laminar flow clean hood void of trace metals contamination and ultra-violet radiation. Upon reception, the samples were designated discrete sample identifiers. An aliquot of each sample was filtered (0.45µm) and these filtrates were stored in a secure, monitored cryofreezer (maintained at a temperature of -80^{o}C) until selenium speciation analysis could be performed via ion chromatography inductively coupled plasma dynamic reaction cell mass spectrometry (IC-ICP-DRC-MS).

2. Sample Preparation

All sample preparation is performed in laminar flow clean hoods known to be free from trace metals contamination. All applied water for dilutions and sample preservatives are monitored for contamination to account for any biases associated with the sample results.

Selenium Speciation Analysis by IC-ICP-DRC-MS Prior to analysis, an aliquot of each sample was filtered with a syringe filter (0.45µm) and injected directly into a sealed autosampler vial. No further sample preparation was performed as any chemical alteration of the samples may shift the equilibrium of the system resulting in changes in speciation ratios.

3. Sample Analysis

All sample analysis is precluded by a minimum of a five-point calibration curve spanning the entire concentration range of interest. Calibration curves are performed at the beginning of each analytical day. All calibration curves, associated with each species of interest, are standardized by linear regression resulting in a response factor. All sample results are **instrument blank corrected** to account for any operational biases associated with the analytical platform.

Prior to sample analysis, all calibration curves are verified using second source standards which are identified as initial calibration verification standards (ICV).

Ongoing instrument performance is identified by the analysis of continuing calibration verification standards (CCV) and continuing calibration blanks (CCB) at a minimal interval of every ten analytical runs.

Selenium Speciation Analysis by IC-ICP-DRC-MS All samples for selenium speciation analysis were analyzed by ion chromatography inductively coupled plasma dynamic reaction cell mass spectrometry (IC-ICP-DRC-MS) on October 15, 2010. An aliquot of each sample is injected onto an anion exchange column and mobilized by a basic (pH > 7) gradient. The eluting selenium species are then introduced into a radio frequency (RF) plasma where energy-transfer processes cause desolvation, atomization, and ionization. The ions are extracted from the plasma through a differentially-pumped vacuum interface and travel through a pressurized chamber (DRC) containing a specific reactive gas which preferentially reacts with interfering ions of the same target mass to charge ratios (m/z). A solid-state detector detects ions transmitted through the mass analyzer and the resulting current is processed by a data handling system.

Retention times for each eluting species are compared to known standards for species identification.

4. Analytical Issues

The overall analyses went very well and no analytical issues were encountered. All quality control parameters associated with these samples were within acceptance limits.

The estimated method detection limits (eMDLs) for selenite, selenate, and selenocyanate are generated from replicate analyses of the lowest standard in the calibration curve. Not all selenium species are present in preparation blanks; therefore, eMDL calculations based on preparation blanks are artificially biased low.

The eMDL for methylseleninic acid and selenomethionine is calculated from the average eMDL of selenite, selenate, and selenocyanate. The calibration does not

contain methylseleninic acid or selenomethionine due to impurities in these standards which would bias the results for other selenium species.

If you have any questions or concerns regarding this report, please feel free to contact me.

Sincerely,

Russell Gerads
Vice President
Applied Speciation and Consulting, LLC

Selenium Speciation Results for Duke Energy
Project Name: Allen - FGD Alternate Fuels Test Burn #1 - Se
Contact: Jay Perkins
LIMS #J10090181

Date: October 17, 2010
Report Generated by: Russell Gerads
Applied Speciation and Consulting, LLC

Sample Results

Sample ID	Se(IV)	Se(VI)	SeCN	MeSe(IV)	SeMe	Unknown Se Species (n)
FGD Purge Eff	13.1	236	ND (<3.0)	ND (<2.4)	ND (<2.4)	0 (0)
BioReactor 1 Inf	5.48	574	ND (<0.75)	ND (<0.60)	ND (<0.60)	0 (0)
BioReactor 2 Eff	0.92	ND (<0.42)	ND (<0.75)	ND (<0.60)	ND (<0.60)	0 (0)
Metals Trip Blk	ND (<0.13)	ND (<0.084)	ND (<0.15)	ND (<0.12)	ND (<0.12)	0 (0)

All results reflect the applied dilution and are reported in µg/L
ND = Not detected at the applied dilution
SeCN = Selenocyanate
MeSe(IV) = Methylseleninic acid
SeMe = Selenomethionine
Unknown Se Species = Total concentration of all unknown Se species observed by IC-ICP-MS
n = number of unknown Se species observed

Selenium Speciation Results for Duke Energy
Project Name: Allen - FGD Alternate Fuels Test Burn #1 - Se
Contact: Jay Perkins
LIMS #J10090181

Date: October 17, 2010
Report Generated by: Russell Gerads
Applied Speciation and Consulting, LLC

Quality Control Summary - Preparation Blank Summary

Analyte (µg/L)	PBW1	PBW2	PBW3	PBW4	Mean	StdDev	eMDL*	eMDL 10x	eMDL 50x	eMDL 200x
Se(IV)	0.000	0.000	0.000	0.000	0.000	0.000	0.013	0.13	0.63	2.5
Se(VI)	0.000	0.000	0.000	0.000	0.000	0.000	0.008	0.084	0.42	1.7
SeCN	0.000	0.000	0.000	0.000	0.000	0.000	0.015	0.15	0.75	3.0
MeSe(IV)	0.000	0.000	0.000	0.000	0.000	0.000	0.012	0.12	0.60	2.4
SeMe	0.000	0.000	0.000	0.000	0.000	0.000	0.012	0.12	0.60	2.4

eMDL = Estimated Method Detection Limit
*Please see narrative regarding eMDL calculations

Quality Control Summary - Certified Reference Materials

Analyte (µg/L)	CRM	True Value	Result	Recovery
Se(IV)	ICV	9.57	9.73	101.7
Se(VI)	ICV	9.48	9.23	97.3
SeCN	ICV	8.92	9.45	105.9
MeSe(IV)	ICV	6.47	6.24	96.4
SeMe	ICV	9.32	10.01	107.4

Selenium Speciation Results for Duke Energy
Project Name: Allen - FGD Alternate Fuels Test Burn #1 - Se
Contact: Jay Perkins
LIMS #J10090181

Date: October 17, 2010
Report Generated by: Russell Gerads
Applied Speciation and Consulting, LLC

Quality Control Summary - Matrix Duplicates

Analyte (µg/L)	Sample ID	Rep 1	Rep 2	Mean	RPD
Se(IV)	Batch QC	ND (<0.63)	0.86	NC	NC
Se(VI)	Batch QC	ND (<0.42)	ND (<0.42)	NC	NC
SeCN	Batch QC	ND (<0.75)	ND (<0.75)	NC	NC
MeSe(IV)	Batch QC	ND (<0.60)	ND (<0.60)	NC	NC
SeMe	Batch QC	ND (<0.60)	ND (<0.60)	NC	NC

ND = Not detected at the applied dilution
NC = Value was not calculated due to one or more concentrations below the eMDL

Quality Control Summary - Matrix Spike/ Matrix Spike Duplicate

Analyte (µg/L)	Sample ID	Spike Conc	MS Result	Recovery	Spike Conc	MSD Result	Recovery	RPD
Se(IV)	Batch QC	278.0	231.4	83.2	278.0	224.3	80.7	3.1
Se(VI)	Batch QC	252.3	222.2	88.1	252.3	217.4	86.2	2.2
SeCN	Batch QC	228.8	200.1	87.5	228.8	200.3	87.6	0.1

CHAIN OF CUSTODY RECORD AND ANALYSIS REQUEST FORM

4

Duke Energy

Duke Energy Analytical Laboratory
Mail Code MGO3A2 (Building 7405)
13339 Hagers Ferry Rd
Huntersville, N. C. 28078
(704) 875-5245
Fax: (704) 875-4349

Analytical Laboratory Use Only

Order # J10090181 | Matrix OTHER | Samples Originating From NC___ SC___

Logged By J. Herman | Date & Time 9/28/10 17:18 | SAMPLE PROGRAM Ground Water___ NPDES___ Drinking Water___ UST___ RCRA Waste___

Vendor AS&C
PO# ISW01.1894

17, 8 (10/12) Cooler Temp (C)

[15]Preserv.: 1=HCl 2=H_2SO_4 3=HNO_3 4=Ice 5=None

[19]Page 1 of 2
DISTRIBUTION
ORIGINAL to LAB,
COPY to CLIENT

Customer must Complete

[1]Project Name: Allen - FGD Alternative Fuels Test Burn # 1 - Se		[2]Phone No:
[2] Client: Don Scruggs, Robbin Jolly, Ray Lidke, Bill Kennedy		[4]Fax No:
[5]Business Unit:	[6]Process:	Mail Code:
[8]Oper. Unit:	[9]Res. Type:	[10]Resp. Center:

MR #

Customer to complete all appropriate non-shaded areas.

Sampling conducted: 2nd and 4th Monday

[16]Analyses Required

LAB USE ONLY [11]Lab ID	Se Speciation Bottle ID	[13]Sample Description or ID (due 10-25)	Date	Time	Signature	[17]Comp.	[18]Grab	Se, speciation - vendor to AS&C (Important to place filled bottle back into both baggies)
2010008548	B069446	FGD Purge Eff	10/8	1018	JEFF WOMACK			1
2010008549	B069901	BioReactor 1 Inf	10/8	0951	Jeremy Dawson			1
2010008550	B069888	BioReactor 2 Eff	10/8	0942	Jeremy Dawson			1
2010008551	B058669	Metals Trip Blk	9/30	1345	K. Harris			1

Customer to complete appropriate columns to right

Customer to sign & date below - fill out from left to right.

1) Relinquished By	Date/Time 10/11/10 11:00	2) Accepted By	Date/Time 10-11-10 1100
3) Relinquished By	Date/Time 10-11-10 1605	4) Accepted By	Date/Time 10-11-10 1605
5) Relinquished By	Date/Time 10-12-10 1030	6) Accepted By:	Date/Time
7) Relinquished By	Date/Time	8) Accepted By:	Date/Time
9) Seal/Locked By	Date/Time 10-12-10 1000	10) Seal/Lock Opened By	Date/Time 10/13/10 9:15 Temp: 0.2°C
11) Seal/Locked By	Date/Time	12) Seal/Lock Opened By	Date/Time

Comments: * Metals=As, Ag, B, Cu, Cr, Ni, Se, Zn

Customer, IMPORTANT!
Please indicate desired turnaround.

10-21

CHAIN OF CUSTODY RECORD AND ANALYSIS REQUEST FORM

4

Duke Energy

Duke Energy Analytical Laboratory
Mail Code MGO3A2 (Building 7405)
13339 Hagers Ferry Rd
Huntersville, N. C. 28078
(704) 875-5245
Fax: (704) 875-4349

Analytical Laboratory Use Only

Order #	Matrix	Samples Originating From
J10090181	OTHER	NC___ SC___

Logged By	Date & Time	SAMPLE PROGRAM
J. Herman	9/28/10 17:18	Ground Water ___ NPDES ___ Drinking Water ___ UST ___ RCRA Waste ___

Vendor AS&C
PO# ISW01.1894

17.8 (10/12)
Cooler Temp (C)

[19]Page 1 of 2
DISTRIBUTION
ORIGINAL to LAB,
COPY to CLIENT

1)Project Name	Allen - FGD Alternative Fuels Test Burn # 1 - Se	2)Phone No:
2) Client:	Don Scruggs, Robbin Jolly, Ray Lidke, Bill Kennedy	4)Fax No:
5)Business Unit:	6)Process:	Mail Code:
8)Oper. Unit:	9)Res. Type:	10)Resp. Center:

MR #

Customer to complete all appropriate non-shaded areas.

[15]Preserv.:1=HCL 2=H_2SO_4 3=HNO_3

[16]Analyses Required

due 10-25

Sampling conducted 2nd and 4th Monday

LAB USE ONLY [11]Lab ID	Se Speciation Bottle ID	[13]Sample Description or ID	Date	Time	Signature	[17]Comp.	[18]Grab	Se, speciation - vendor to ASC (Important to place filled bottle back into both baggies) ✓
2010008548	B06946	FGD Purge Eff	10/8	1018	JEFF Womack			1
2010008549	B0690	BioReactor 1 Inf	10/8	0951	Jeremy Dawson			1
2010008550	B06988	BioReactor 2 Eff	10/8	0942	Jeremy Dawson			1
2010008551	B05869	Metals Trip Blk	9/30	1345	[signature]			1

*Customer to sign & date below - fill out from left to right

1) Relinquished By	Date/Time	2) Accepted By	Date/Time
[signature]	10/11/10 11:00	[signature]	10-11-10 1100
3) Relinquished By [signature]	10-11-10 1605	4) Accepted By [signature]	10-11-10 1605
5)Relinquished By [signature]	10-12-10 10:??	6)Accepted By:	
7)Relinquished By		8)Accepted By:	
9)Seal/Locked By [signature]	10-12-10 1000	10) Seal/Lock Opened By	
11)Seal/Locked By		12)Seal/Lock Opened By	

Comments
* Metals=As, Ag, B, Cu, Cr, Ni, Se, Zn

Customer IMPORTANT! Please indicate desired turnaround

10-21